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XXII.

CONTRIBUTIONS FROM THE CHEMICAL LABORATORY OF
HARVARD COLLEGE.ON THE BEHAVIOR OF SOUND AND DECAYED
WOOD AT HIGH TEMPERATURES.

BY HENRY B. HILL AND ARTHUR M. COMEY.

SOME time ago, at the request of Mr. Alexander Agassiz, President of the Calumet and Hecla Mining Company, one of us studied the behavior of sound and decayed wood at high temperatures, in order to determine the minimum point of ignition of decayed wood, and the lowest temperature at which volatile decomposition products were evolved. Through the kindness of Mr. Agassiz, we are able to present to the Academy the result of the investigation.

I. ON THE MINIMUM POINT OF IGNITION OF
DECAYED WOOD.

BY HENRY B. HILL.

After a series of preliminary experiments to determine the conditions most favorable to ignition and the form of apparatus best adapted to the purpose, I had made a cylindrical air-bath of sheet-iron fitted with a suitable aperture for the admission of air and a short vertical draught pipe, which was sufficient to maintain a free circulation of air when the bath was heated. A horizontal perforated shelf was fixed in the bath, parallel to the axis of the cylinder, and in order to prevent direct radiation or conduction of heat this shelf was covered with a double layer of asbestos paper so arranged as to leave an air space of about five millimeters between the two layers.

Above the shelf was placed the bulb of the thermometer, and close beside it the sample to be tested. The bulb of the thermometer was further protected from the heat radiated from the sample shortly before ignition by a double screen of asbestos paper. On the upper side of the air bath was cut a rectangular opening closed with a movable

mica plate, through which the samples could be introduced and the ignition noted.

In order to avoid loss of heat by radiation the bath was surrounded by a loose envelope of asbestos paper, which allowed the hot gases from the lamps beneath to circulate about the bath. For determining temperatures below 200° I used a small mercurial thermometer; for temperatures above 200° , an air thermometer with constant air manometer, on the plan described some time since by Professor J. P. Cooke.* This thermometer proved to be extremely convenient, and its indications agreed closely with those of the mercurial thermometer in the neighborhood of 200° .

In preliminary trials, which I made with sound pine wood, I soon found that the ignition point was greatly affected by the way in which the sample was heated. If the temperature was slowly raised, the wood was thoroughly carbonized before the ignition point was reached, and the observed ignition point was merely that of charcoal. Thus four samples of pine-wood, slowly heated, ignited when the thermometer reached 405° , 407° , 415° , and 417° , while freshly burned pine charcoal ignited at 403° , 405° , and 408° , at successive trials. Pieces of the same wood (when the bath was quickly heated) ignited at 360° , 365° , and 372° , and when dropped into the heated bath they also took fire at lower temperature.

In order to determine the lowest possible point of ignition, it was therefore evidently necessary to make successive trials, in each case introducing the sample into the bath heated to a definite temperature, and lowering this temperature a few degrees at a time until the sample failed to ignite. Small pieces of wood (0.5 gram) usually ignited, if at all, within fifteen or twenty minutes; larger pieces (15 grams) sometimes required heating for an hour or longer.

The samples of wood which I examined were all portions of timbers from the Calumet and Hecla Mine, and varied greatly in physical properties. They were:—

A.	Pine	from the 14th level about 5 years in the mine.				
B.	"	"	7	"	6	"
C.	"	"	6	"	7	"
D.	"	"	6	"	8	"
E.	"	"	7	"	8	"
F.	Hemlock	"	9	"	5	"
G.	"	"	7	"	7	"
H.	"	"	6	"	9	"
I.	"	"	12	"	10	"

* These Proceedings, XVII. 22.

From these I selected a variety of samples differing most widely in their physical properties and appearance, and examined with most care those portions which were most decayed. While it was evident that a large variation in the size of the piece of wood taken produced a corresponding variation in the ignition point, it seemed to me advisable to determine at the outset the comparative points of ignition of the various portions, using in each case pieces of approximately the same size, and subsequently to determine the variation caused by taking larger pieces. In the following determinations pieces of wood measuring about 1.6 cubic centimeters were used, and since the wood was very light they weighed about half a gram each.

A. Inside Wood, sound.

°
285 failed.
300 ignited.
300 “

B. Inside Wood, sound.

°
300 failed.
305 “
318 “
330 ignited.

C. (a.) Sap-wood.

°
252 ignited.
250 “
252 “
250 “
245 “
243 “
239 “
232 failed.

C. (b.) Inside.

°
248 ignited.
243 “
242 “
238 “
230 failed.

D. (a.) Sap-wood, decayed fibrous.

°
245 ignited.
243 “
238 “
238 “
233 “
232 “
230 “
226 failed.

D. (b.) Sap-wood, decayed fibrous.

°
228 ignited.
226 “
222 “
218 “
218 “
215 failed.
(c.) 238 ignited.
224 failed.
(d.) 235 ignited.
227 failed.

E. Sap-wood, decayed.

^o
 260 ignited.
 250 “
 245 “
 245 “
 239 “
 235 “
 233 “
 233 “
 230 “
230 “
 228 failed.

F. Inside Wood, sound.

^o
285 ignited.
 275 failed.

G. (a.) Sap-wood, decayed.

^o
 255 ignited.
 247 “
 240 “
 235 “
 230 “
223 “
 219 failed.

G. (b.) Inside Wood, decayed.

^o
 233 ignited.
 230 “
 223 “
220 “
 212 failed.
 218 ignited.
 215 “
210 “
 205 failed.
208 ignited.
 205 failed.

G. (c.) Inside Wood, decayed.

^o
 228 ignited.
 224 “
220 “
 215 failed.

H. (a.) Inside Wood, sound.

^o
 315 ignited.
 303 “
 297 “
 288 “
 278 “
255 “
 245 failed.

H. (b.) Sap-wood.

^o
235 ignited.
 202 failed.

I. Sap-wood.

^o
 215 failed.
 225 ignited.
220 “
 215 failed.

The following table gives the lowest observed ignition point of each sample tested, together with the temperature, at which the wood failed to ignite.

	A.	B.	C. (a.)	C. (b.)	D. (a.)	D. (b.)	D. (c.)	D. (d.)
Ignited,	300°	330°	239°	238°	230°	218°	238°	235°
Failed,	285	318	232	230	226	215	224	227

	E.	F.	G. (a.)	G. (b.)	G. (c.)	H. (a.)	H. (b.)	I.
Ignited,	230°	285°	223°	208°	220°	255°	235°	220°
Failed,	228	275	219	205	215	245	232	215

It will be seen that the ignition point of the pine wood varied from 330° in the comparatively sound portion of B to 218° in the decayed fibrous portions of D. Among the samples of hemlock, F, which was quite sound, ignited at 285°, while a portion of G, which though compact was completely softened by decay, ignited at 208°.

It is hardly necessary to say that the actual temperature of the sample at the time of ignition was very much above that of the bath, so that the observed temperatures are those at which oxidation became sufficiently rapid to heat the sample to self-supporting combustion. The great differences in the temperatures which were found necessary to effect this rapid oxidation seemed to be conditioned chiefly by the degree of porosity, the more porous woods giving free access to the oxygen of the air and at the same time conducting heat, but slowly, so that their temperature was rapidly raised to the point of ignition by the oxidation started at lower temperature.

In several cases I watched more closely the progress of the oxidation by introducing into the bath a second thermometer, whose small bulb was brought near the sample of wood. For some time the two thermometers showed the same temperature, then, the temperature of the bath remaining constant, the second thermometer began to rise slowly at first, and afterwards so rapidly that it had to be withdrawn from the bath before the sample actually took fire.

Since it seemed probable that the oxidation of a porous wood would not materially be retarded by increasing its volume, while the loss of heat by conduction and radiation must be diminished, I proceeded to test the effect of increasing the size of the sample taken upon the ignition point. For this purpose I selected that portion of G which had already shown the lowest ignition point. On repeating the determination with small bits of the wood, it gave a somewhat lower temperature than that before obtained, and by increasing the size of the sample

it was found that the ignition point was materially lowered. The specific gravity of the wood obtained by measurement varied somewhat, but was not far from 0.3.

Weight.	Approximate Volume.	Temperature.
Grams.	c. c.	°
0.5	1.7	208 ignited.
0.5	1.7	206 “
0.5	1.7	204 “
0.5	1.7	202 failed.
3.0	10.0	200 ignited.
3.0	10.0	197 “
3.0	10.0	195 “
3.0	10.0	191 “
3.0	10.0	186 “
3.0	10.0	181 “
3.0	10.0	176 failed.
9.0	30.0	175 ignited.
9.0	30.0	170 failed.
18.0	60.0	171 ignited.
16.8	56.0	166 “
15.5	51.7	163 “
15.8	52.7	158 “
17.0	56.7	154 failed.
13.0	43.3	154 “

It will be seen that the point of ignition with 0.5 gram of the wood was 204°; with 3 grams, 181°; with 9 grams, 175°; and with 16 grams, 158°. The dimensions of my air-bath were such that no larger pieces of the wood could be used, and, moreover, from the material at my command it was impossible to obtain larger compact pieces of this particular sort of wood. It would seem probable, however, that with larger masses of this porous wood ignition could easily be started by a much lower initial temperature.

II. ON THE DECOMPOSITION OF WOOD AT HIGH TEMPERATURES.

BY HENRY B. HILL AND ARTHUR M. COMEY.

ALTHOUGH preliminary experiments had shown that gaseous products of decomposition were formed from wood in considerable quantity only at comparatively high temperatures, it seemed to us of interest to follow the reaction more closely, and to determine quantitatively the extent of the decomposition at various temperatures. Since the investigation was originally undertaken to determine whether this decomposition had any practical importance, it also seemed advisable to select the conditions usually met with in practice, and to heat the wood in a slow current of air rather than *in vacuo* or in a current of an inert gas. Since volatile products were formed in but small quantities, it was useless to attempt any separation or identification of them other than the discrimination between the carbonic dioxide directly formed and the carbon volatilized in other forms. The carbonic dioxide we determined by absorption in a standard solution of baric hydrate, and the volatile carbon by the same method after combustion with copper oxide. The method we employed was as follows. A definite weight of wood in the form of small fragments was introduced into a glass tube and heated in an air-bath to the requisite temperature. Through the tube was then drawn a slow current of air, which had been freed from volatile carbon compounds and carbonic dioxide by passing over red-hot copper oxide and washing with potassic hydrate. A wash-bottle filled with a concentrated solution of baric hydrate, through which the air passed in entering the tube, served to prove the complete removal of the carbonic dioxide. On leaving the tube, the air passed through a measured quantity of a baric hydrate solution of known strength, and the complete absorption of the carbonic dioxide was proved as before by means of a wash-bottle containing a stronger solution of baric hydrate. The air thus freed from carbonic dioxide was led over a small roll of copper gauze heated to redness, and the carbonic dioxide thus formed absorbed as before in a measured quantity of a standard solution of baric hydrate, while a second wash-bottle with baric hydrate served to control the absorption. After the lapse of a sufficient time, the amount of carbonic dioxide directly formed, and that formed by the combustion of the volatile carbon compounds was determined by titration of the baric hydrate solutions with oxalic

acid, phenolphthalein being used as an indicator. The oxalic acid used contained 2.8636 grams of pure crystallized oxalic acid to the litre, and one centimeter of the solution was therefore equivalent to one milligram of carbonic dioxide. While it was evident that any volatile acids formed and carried into the baric hydrate would be counted as carbonic dioxide, direct experiments convinced us that the error introduced in this way, even at the higher temperatures employed, was inappreciable. For convenience in the comparison of our results we have in each case calculated the number of milligrams of carbonic dioxide directly and indirectly formed in one hour from each gram of wood.

Our first series of experiments was made with sound pine wood.

- I. 2 grm. of wood when heated for 1 hour at 220° gave 13.35 mg. direct and 3.5 mg. indirect CO₂.
- II. 2 grm. of wood when heated for 2 hours at 200° gave 10.6 mg. direct and 4.1 mg. indirect CO₂.
- III. 2 grm. of wood when heated for 2 hours at 180° gave 3 mg. direct and 1.8 mg. indirect CO₂.
- IV. 2 grm. of wood when heated for 3 hours at 162° gave 2.6 mg. direct and 2.1 mg. indirect CO₂.
- V. 2 grm. of wood when heated for 5 hours at 143° gave 1.7 mg. direct and 2 mg. indirect CO₂.
- VI. 2 grm. of wood when heated for 7 hours at 123° gave 1.3 mg. direct and 2.8 mg. indirect CO₂.

Sound Pine Wood.

	Temperature.	Milligrams of CO ₂ in one Hour for each Gram of Wood.	
		Direct.	Indirect.
	°		
I.	220	6.67	1.75
II.	200	2.65	1.03
III.	180	0.75	0.45
IV.	162	0.43	0.35
V.	143	0.17	0.20
VI.	123	0.09	0.20

In order to determine whether the indirect carbonic dioxide was formed wholly or in part from the volatile constituents of the wood, which were volatilized without decomposition, two successive determinations were made at the same temperature, with the same sample of wood and the products formed determined as before.

- I. 2 grm. of sound pine wood heated for 2 hours at 200° gave 11 mg. direct and 4 mg. indirect CO_2 .
- II. The same wood heated for 2 hours more at 200° gave 9 mg. direct, and 3 mg. indirect CO_2 .

	$^{\circ}$	Direct.	Indirect.
I.	200	2.75	1.00
II.	200	2.25*	0.75*

Although the decomposition was less rapid during the second heating, the ratio between the direct and the indirect carbonic dioxide remained essentially unchanged.

Before proceeding further, we were interested to determine whether a pure form of cellulose would yield similar results. We therefore made three experiments with washed Swedish filter paper; and found that a similar, though much more slow, decomposition ensued.

- I. 3 grm. washed Swedish filter paper heated for $3\frac{1}{2}$ hours at 210° gave 4.1 mg. direct and 2.3 mg. indirect CO_2 .
- II. 3 grm. washed Swedish filter paper heated for $4\frac{1}{2}$ hours at 180° gave 1.5 mg. direct and 2.5 mg. indirect CO_2 .
- III. 3 grm. washed Swedish filter paper heated for 7 hours at 160° gave 0.3 mg. direct and 2.3 mg. indirect CO_2 .

Swedish Filter Paper.

	Temperature.	Milligrams of CO_2 in one Hour for each Gram.	
	$^{\circ}$	Direct.	Indirect.
I.	210	0.41	0.23
II.	180	0.11	0.19
III.	160	0.01	0.10

With decayed pine wood, a portion of sample D mentioned in the preceding paper, we obtained the following results:—

- I. 1.5 grm. of the wood when heated for 1 hour at 196° gave 12 mg. direct and 4.3 mg. indirect CO_2 .
- II. 1.5 grm. of the wood when heated for $1\frac{1}{4}$ hours at 180° gave 10.6 mg. direct and 4.3 mg. indirect CO_2 .

* Referred to original weight of air-dried wood.

- III. 1.5 grm. of the wood when heated for $1\frac{1}{4}$ hours at 158° gave 4.6 mg. direct and 1.9 mg. indirect CO_2 .
- IV. 1.5 grm. of the wood when heated for $3\frac{3}{4}$ hours at 140° gave 6.1 mg. direct and 3.5 mg. indirect CO_2 .
- V. 1.5 grm. of the wood when heated for 5 hours at 120° gave 3 mg. direct and 4.2 mg. indirect CO_2 .
- VI. 1.5 grm. of the wood when heated for 6 hours at 100° gave 2.9 mg. direct and 4.6 mg. indirect CO_2 .

Decayed Pine Wood.

	Temperature.	Milligrams of CO_2 in one Hour for each Gram of Wood.	
		Direct.	Indirect.
	$^{\circ}$		
I.	196	8.00	2.87
II.	180	5.65	2.29
III.	158	2.45	1.01
IV.	140	1.08	0.59
V.	120	0.40	0.56
VI.	100	0.32	0.51

We have made no experiments with sound hemlock, but with decayed hemlock we have obtained the following results. The wood taken was from sample G used in the preceding paper.

- I. 1.5 grm. of the wood when heated for 1 hour at 195° gave 19.3 mg. direct and 7.8 mg. indirect CO_2 .
- II. 1.5 grm. of the wood when heated for $1\frac{1}{8}$ hours at 180° gave 16.6 mg. direct and 4.6 mg. indirect CO_2 .
- III. 1.5 grm. of the wood when heated for 1 hour at 160° gave 8.8 mg. direct and 2.7 mg. indirect CO_2 .
- IV. 1.5 grm. of the wood when heated for 1 hour at 140° gave 3 mg. direct and 0.7 mg. indirect CO_2 .
- V. 1.5 grm. of the wood when heated for $1\frac{1}{2}$ hours at 120° gave 1.7 mg. direct and 0.5 mg. indirect CO_2 .
- VI. 1.5 grm. of the wood when heated for 2 hours at 100° gave 1.5 mg. direct and 0.5 mg. indirect CO_2 .

Decayed Hemlock Wood.

	Temperature. °	Milligrams of CO ₂ in one Hour for each Gram of Wood.	
		Direct.	Indirect.
I.	195	12.87	5.20
II.	180	9.49	2.63
III.	160	5.87	1.80
IV.	140	2.00	0.47
V.	120	0.76	0.22
VI.	100	0.50	0.16